

EXHIBIT A

DOCKET NO.: 441-06/RD02036 US

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re Application of:

Applicants: Leo Zhaoqing Liu and Christian Priou

Confirmation No.: 6545

Application No.: 10/607,079

Examiner: White, Everett NMN

Filing Date: June 25, 2003

Art Unit: 1623

For: GRAFTING POLYMERIZATION OF GUAR AND OTHER POLYSACCHARIDES BY ELECTRON BEAMS

Declaration of Dr. Leo Zhaoqing Liu

I, Leo Zhaoqing Liu, declare as follows:

1. I am an Inventor of United States Patent Application 10/607,079, filed June 25, 2003.
2. I obtained my Bachelor of Science and Master of Science degrees from Peking (Beijing) University. I obtained my Ph.D. in Chemistry from the University of British Columbia. I have been a research Assistant at the University of Saskatchewan, a Teaching and Research Assistant at the University of British Columbia, a Postdoctoral Research Fellow at McGill University and a Postdoctoral Research Scientist at the University of Toronto. From 1998-2001, I was employed by Rhodia Canada, Inc., as Senior Chemist. From 2001-2008, I was employed by Rhodia, Inc., initially as a Staff Scientist and then as Senior Staff Scientist. I am now Manager and Senior Staff Scientist in the Rhodia Research & Technology Center of Rhodia China Co., Ltd., located in Shanghai, China.
3. I am familiar with United States Patent Application 10/607,079, including pending claims 21-28.

4. Applicants' invention as claimed in claim 21 is to a method for grafting an unsaturated monomer onto a polysaccharide comprising the steps of:
 - (1) forming a mixture comprised of an unsaturated monomer and a water soluble or water dispersible polysaccharide;
 - (2) drying the mixture; and
 - (3) irradiating the mixture with an amount of electron beam radiation sufficient to form an unsaturated monomer-water soluble or water dispersible polysaccharide graft copolymer, wherein the graft copolymer is depolymerized to a molecular weight lower than the molecular weight of the ungrafted polysaccharide, and the copolymer has a molecular weight of no more than 700,000 Daltons.
5. The remaining claims 22-28 are also directed to the method claimed in claim 21.
6. Included in the claimed method is a step 2 of drying the mixture after the step of forming the mixture and before the step of irradiating the mixture.
7. Several paragraphs in the Application disclose the drying step and the occurrence of the drying step after the step of forming the mixture and before the step of irradiating the mixture.
8. This disclosure is provided in paragraph [047] which reads as follows:

Guar powder was suspended in acetone, and then mixed with either vinyl phosphonic acid (VPA) or methacrylamidopropyltrimethylammonium chloride (MAPTAC) solution at a 10:1 ratio of guar to the respective monomer. *The mixture was then dried in a vacuum* and put into a plastic vial with its weight within the penetration range of the electron beam. The samples were then placed on a tray carried by an endless conveyor into a radiation chamber. The samples were irradiated by electron beam generated by 4.5 MeV generator operating at a 15 milliamps beam current at the top surface of the tray. The desired dose was obtained by adjusting the linear velocity of the conveyor.
9. Support for the drying step, prior mixing step and subsequent irradiation step is also provided by paragraph [052] which reads as follows:

Hydroxypropyl guar, available from Rhodia, Inc., in Cranbury, New Jersey, as Jaguar 8000, 50 parts was mixed with methacrylamidopropyltrimethylammonium chloride (MAPTAC, 50% in

water), 15 parts and methanol 15 parts. *The wet mixture was then dried in a vacuum oven at 30-40°C. The dried powder was then packed in a plastic bag with thickness less than 3 cm. The irradiation was done as described in Example 1 at a dose of 3.8 Mrad. The residual amount of non-reacted MAPTAC was analyzed by HPLC to be 0.39% in the sample (that is 97% conversion). The irradiated sample was then sprayed with 5 grams of 10% sodium metabisulfite solution in 1:1 water/methanol and then cured in a vacuum oven at 65-70°C for two (2) hours. The residual monomer was analyzed again to be 440 ppm.*

10. Additional support is provided by paragraph [055] which reads as follows:
Hydroxyethylcellulose, available from Dow as Cellosize HEC QP 100M-H was sprayed with 50% MAPTAC solution at the ratios of the active components shown in Table 4, and then thoroughly mixed. The MAPTAC-swollen cellulose was then air-dried and ground in to powder for easy handling. The irradiation and the post-treatment were done according to the procedure described in Example 2 with the dose shown in Table 4. The residual MAPTAC was measured by HPLC analysis after the irradiation (Table 4) and after further treatment (Table 5). The molecular weight was determined for selective samples (Table 6). Little or no homopolymer of MAPTAC was detected by the GPC analysis. The grafted polymer was isolated from aqueous methanol solution by precipitating with acetone. Colloid titration of the isolated polymer indicated more than 85% of the MAPTAC was attached to hydroxyethylcellulose.
11. Paragraphs [047], [052], and [055] clearly disclose a drying step (2) between the formation of the mixture in step (1) and the irradiation of the mixture in step (3).
12. I have conducted a series of tests to determine more precisely the effect of the drying step on the method disclosed and claimed in my application. These tests included a drying step in which the initial mixture, produced according to step 1 of the claimed method, was dried to moisture contents of between about 0.74% and 30.2% according to step 2 of the claimed method. The dried mixtures were treated according to step 3 of the claimed process and were measured in terms of the percentage conversion of the mixture to the graft copolymer at different concentrations of electron beam radiation. The data obtained in these tests are set forth in Tables 1-3 which includes a three dimensional graph of the data for each of Tables 1-3. The graphs show for each test the moisture content, the dose of electron beam radiation, and the rate of conversion of the mixture into a polysaccharide graft copolymer.

13. On the basis of the results submitted in Tables 1-3, I determined that for lower doses of electron beam radiation the optimum range for drying the mixture is to a moisture content of 5-20%. Drying the mixture to 30% moisture content at which the dried mixture felt dry still improved the efficiency of the claimed method. The drying step resulted in a higher rate of conversion of the mixture into a polysaccharide graft copolymer. The absence of a drying step also had the disadvantage that a greater amount of electron beam radiation was needed to accomplish the formation of the polysaccharide graft copolymer to the same degree. In addition, the product resulting from the drying step has the advantage that it is generally easier to handle than the product without the drying step.
14. Drying the mixture to a moisture content below 5% will still improve the efficiency of the claimed method. It should be noted that excessive drying, i.e. so that the material contained no moisture, was neither necessary nor desirable as the completely dry mixture could pose a hazard if further subjected to electron beam radiation.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such false statements may jeopardize the validity of Application No. 10/607,079 or any patent issued thereon.

Dated: June 3, 2009



Leo Zhaoqing Liu, Ph.D.

Table 1

% H ₂ O	Dose	c	ppm	%Conversion
3.61	0.5	0.963	263.8	28.95
	1	0.958	215.7	41.60
	3	0.957	129.2	64.98
	5	0.963	97.27	73.80
	10	0.963	65.13	82.46
	15	0.957	49.98	86.45
10.4	0.5	0.861	78.4	74.59
	1	0.891	47.9	85.00
	3	0.894	11.78	96.32
	5	0.894	4.28	98.66
	10	0.896	1.91	99.41
	15	0.885	0.803	99.75
13.3	0.5	0.855	36.58	87.66
	1	0.866	15.09	94.98
	3	0.866	6.69	97.77
	5	0.857	1.32	99.56
	10	0.853	0.377	99.87
	15	0.861	0.3	99.90
19	0.5	0.797	129.6	49.81
	1	0.809	34.2	86.95
	3	0.805	3.67	98.59
	5	0.804	1.41	99.46
	10	0.807	0.138	99.95
	15	0.786	0	100.00
25.8	0.5	0.742	188.42	14.44
	1	0.739	167.1	23.82
	3	0.739	32.06	85.38
	5	0.756	12.26	94.54
	10	0.769	0.167	99.93
	15	0.737	0.692	99.68

% H ₂ O	Dose	c	ppm	%Conversion
29.6	0.5	0.698	234.5	0.00
	1	0.7	212.3	0.00
	3	0.703	76.97	61.12
	5	0.703	31.37	84.15
	10	0.702	0.717	99.64
	15	0.696	0.143	99.93

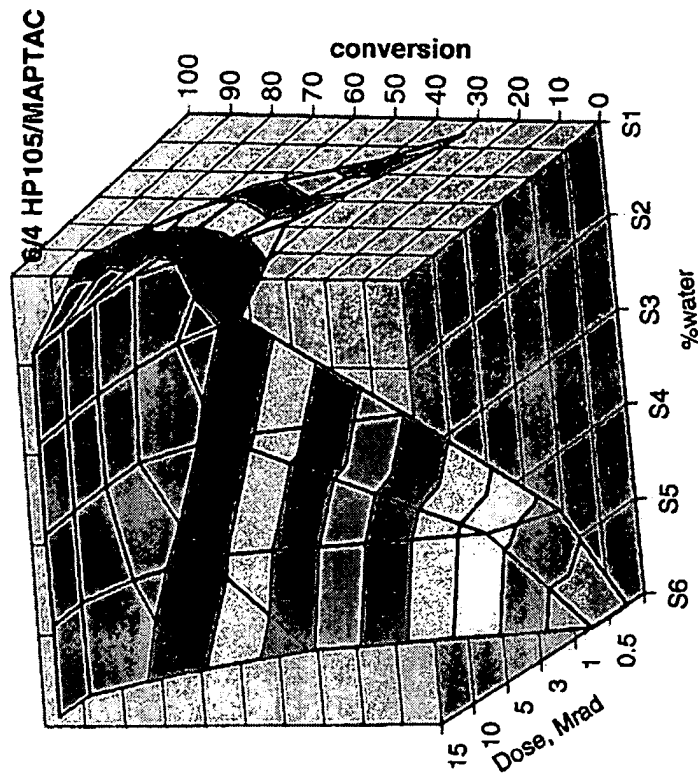
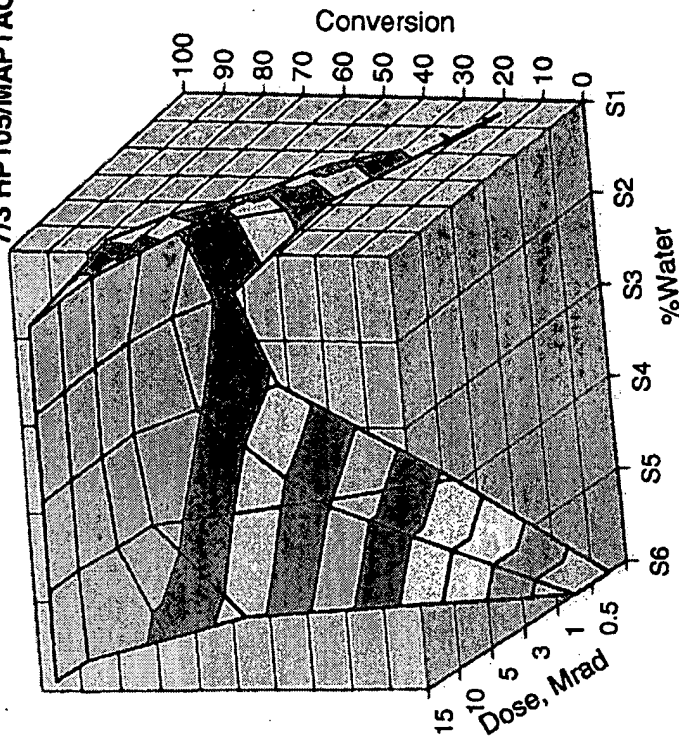


Table 2

% H ₂ O	Dose	c	ppm	%Conversion
2.45	0.5	0.972	237.5	16.51
	1	0.965	225.8	20.04
	3	0.974	160.3	43.76
	5	0.97	135.1	52.41
	10	0.97	86.3	69.60
	15	0.975	61.3	78.52
10.4	0.5		82.77	61.40
	1		94.93	88.64
	3		97.21	93.75
	5		99.39	98.63
	10		99.87	99.71
	15		99.88	99.73
15.6	0.5	0.839	26.18	87.68
	1	0.841	10.23	95.20
	3	0.841	3	98.59
	5	0.823	0.931	99.55
	10	0.837	0.245	99.88
	15	0.829	0.17	99.92
19.8	0.5	0.794	41.68	78.18
	1	0.827	18.03	90.94
	3	0.799	1.87	99.03
	5	0.796	0.85	99.56
	10	0.793	0.134	99.93
	15	0.777	0.123	99.93
25.6	0.5	0.74	104.5	36.73
	1	0.742	61.4	62.93
	3	0.739	6.2	96.24
	5	0.712	4.04	97.46
	10	0.74	0.0865	99.95
	15	0.741	0.066	99.96

7/3 HP105/MAPTAC



% H ₂ O	Dose	c	ppm	%Conversion
30.2	0.5	0.696	162.7	0.00
	1	0.693	171.7	0.00
	3	0.692	36.3	74.95
	5	0.695	18.8	87.08
	10	0.687	1.2	99.17
	15	0.689	0.0497	99.97

Table 3

% H ₂ O	Dose	c	ppm	%Conversion
0.74	0.5	0.986	93.7	67.43
	1	0.99	98.69	65.83
	3	0.987	76.06	73.59
	5	0.991	71.61	75.23
	10	0.981	47.1	83.55
	15	0.975	30.02	89.45
8.59	0.5	0.906	71.05	71.40
	1	0.894	58.33	76.21
	3	0.914	17.97	92.83
	5	0.913	12.26	95.10
	10	0.905	4.34	98.25
	15	0.91	0.41	99.84
15.6	0.5	0.843	6.23	97.08
	1	0.841	2.5	98.83
	3	0.841	1.3	99.39
	5	0.842	0.251	99.88
	10	0.823	0.093	99.96
	15	0.809	0.0948	99.95
19.2	0.5	0.806	7.69	96.06
	1	0.801	2.6	98.66
	3	0.805	0.378	99.81
	5	0.798	0.133	99.93
	10	0.799	0.189	99.90
	15	0.789	0.057	99.97
22.4	0.5	0.762	6.51	96.33
	1	0.772	4.34	97.59
	3	0.773	0.577	99.68
	5	0.773	0.133	99.93
	10	0.769	0.0283	99.98
	15	0.769	0.065	99.96

% H ₂ O	Dose	c	ppm	%Conversion
29.6	0.5	0.694	31.12	78.83
	1	0.704	23.25	84.41
	3	0.693	7.11	95.16
	5	0.689	1.05	99.28
	10	0.703	0.0287	99.98
	15	0.7	0.0269	99.98

